

**Miller, James (ASRC)**

683887

---

**From:** STIC-ILL  
**Sent:** Thursday, April 30, 2009 10:53 AM  
**To:** Miller, James (ASRC)  
**Subject:** FW: Reference Delivery Request, Case/Application No.: 10/759,678

---

**From:** STIC-EIC1700  
**Sent:** Thursday, April 30, 2009 10:47 AM  
**To:** STIC-ILL  
**Subject:** FW: Reference Delivery Request, Case/Application No.: 10/759,678

---

**From:** PAUL MARCANTONI [mailto:Paul.Marcantoni@uspto.gov]  
**Sent:** Wednesday, April 29, 2009 7:14 PM  
**To:** STIC-EIC1700  
**Subject:** Reference Delivery Request, Case/Application No.: 10/759,678

Requester: **PAUL MARCANTONI (P/1793)**  
Art Unit: **GROUP ART UNIT 1793**  
Employee Number: **68189**  
Office Location: **REM 9B17**  
Phone Number: **(571)272-1373**  
Case/Application number: **10/759,678**

Author: **B.E. Scheetz; E.L. White; D Wolfe Confer; D.M. Roy**  
Article or Chapter Title: **"Effect of mix rheology, admixtures and salts on physical and mechanical properties of hardened cement pastes"**  
Journal or Book Title: **Int'l Congr. Chem. Cem. [Proc.], 7th (1980) vol.3, VI/170-175**  
Volume and Issue Number: **Vol.3, VI/170-VI/175**  
Pages: **170-175**  
Publication Date: **1980**  
ISSN/ISBN:  
Other Identifying Infor.: Edition, PMID:  
Remarks:

**AN 98:39663 CA Chem Abstracts STN "AN" number; Also, these gentlemen were from Pennsylvania State University Materials Research lab. This article is for Board of Appeals as well.**

4/30/2009

AS 4/30  
AT 4/30

## VI-170

# Effect of mix rheology, admixtures and salts upon physical and mechanical properties of hardened cement pastes

## *Action combinée des adjuvants et des additions de sel sur les propriétés physiques et mécaniques des pâtes de ciment durcies*

B.E. SCHEETZ, Research Associate,  
 E.L. WHITE, Research Associate,  
 D. WOLFE-CONFER, Senior Research Technician, and  
 D.M. ROY, Professor, Materials Research Laboratory, The Pennsylvania State University, University Park,  
 Pennsylvania 16802, U.S.A.

**SUMMARY:** The interrelationships between the rheological properties of ordinary cement slurries and viscous pastes and the mechanical and physical properties of their hardened composites have been investigated. The cement slurries, made from an ASTM Type I and an API Class C cement, were modified by addition of two superplasticizing admixtures, a sulfonated melamine formaldehyde condensate (A14) and a sulfonated naphthalene formaldehyde condensate (A3). The interactions between the admixtures and brines are important since these composites may be exposed to brines at elevated temperatures (geothermal applications) and to bedded evaporite deposits (in a possible nuclear waste disposal site).

Rheological properties of cement slurries were measured at different shear rates to explore properties related to pumpability in the mixes. The shear stress for both the Type I and the Class C cement slurries was found to decrease with increasing superplasticizer concentration and with increasing salt content at a 1% admixture concentration. However, the rheological properties of both cement pastes were very erratic below 15 wt/o NaCl. In the presence of 25 wt/o NaCl the fluidities of the pastes were 2 to 3 times greater at  $\leq 1\%$  superplasticizer concentrations but changed rapidly in contact with additional amounts of either superplasticizer.

The setting times for the Type I cement slurries systematically increased with increased admixture concentrations; however, the Class C cement had the opposite trend. The possible reason for these behaviors are discussed. The bulk densities for all specimens were measured and variations with curing time, admixture content, and nature of mixing waters were discussed.

The microhardness measurement was utilized as a criterion to determine the strength of a specimen. Generally, the hardness increased with increasing curing time. Variation in hardness values for samples prepared from various cements and mixing water (with or without additives) were also discussed. A very distinct difference in hardness between the samples prepared with deionized water and admixture and saturated sodium chloride and admixture exists. The deionized water prepared samples were fully 40% stronger than the salt-admixture prepared samples.

### RESUME

Les relations entre les propriétés rhéologiques de barbotines de ciments ordinaires ainsi que de pâtes visqueuses et les propriétés mécaniques et physiques de leurs composés durcis ont été étudiées. Les barbotines de ciment, préparées à partir de ciments ASTM type I et API Classe C, furent modifiées par l'ajout de deux adjuvants superplastifiants, un condensé sulphoné de formaldéhyde de mélamine (A14) et un condensé sulphoné de formaldéhyde de naphthalène (A3). Les interactions entre les adjuvants et les saumures sont importantes vu que ces composites peuvent être exposés à des saumures à des températures élevées (applications géothermales) ainsi qu'à des dépôts d'évaporite (dans un site éventuel de stockage de déchets radioactifs).

Les propriétés rhéologiques des barbotines de ciment furent mesurées à différentes vitesses de cisaillement afin d'explorer les propriétés en rapport avec l'aptitude au pompage des mélanges. La force de cisaillement pour les pâtes du Type I et de la Classe C diminue lorsque la concentration de superplastifiant augmente ainsi qu'avec une augmentation du taux de sel lorsque l'on utilise 1% d'adjuvants. Toutefois, les propriétés rhéologiques des deux pâtes de ciment deviennent erratiques lorsque l'on utilise moins de 15% (en poids) de NaCl. En présence de 25% (en poids) de NaCl, les fluidités des pâtes sont 2 à 3 fois supérieures pour des concentrations de superplastifiants  $\leq 1\%$ , mais changent rapidement au contact d'ajouts supplémentaires d'un des deux superplastifiants.

Le temps de prise des pâtes de ciment du Type I est systématiquement allongé lorsque l'on augmente la concentration des adjuvants contrairement au ciment de la Classe C. Les raisons possibles pour ces phénomènes sont exposées. Les densités de tous échantillons furent mesurées et leurs variations avec le temps de prise, le taux d'adjuvants et la nature des eaux de mixage sont rapportées.

La microdureté fut utilisée comme critère pour déterminer la résistance d'un échantillon. En général, la microdureté augmente avec le temps de prise. La variation de la dureté d'échantillons préparés à partir de différents ciments et eaux de mixage (avec ou sans additifs) est aussi rapportée. Il existe une différence très nette entre la dureté d'échantillons préparés avec de l'eau dé-ionisée et des adjuvants et celle d'échantillons préparés avec de l'eau saturée en chlorure de sodium et des adjuvants. Les échantillons préparés avec de l'eau dé-ionisée sont 40% plus résistants que ceux préparés avec l'eau saturée en NaCl.

## INTRODUCTION

Effective sealing of penetrations into a nuclear waste repository (1), or through rock strata made for the purpose of extracting geothermal energy (2,3) will demand the development of cementitious composite materials for closure of the holes, or for cementing of the casings. The composites will need to possess long-term stability (4) coupled with ease of emplacement. Fundamental to the geothermal applications is the exposure of these cementitious composites to mineral-laden brines at elevated temperatures (2,3); while, for the nuclear waste disposal application, the most conceptually advanced repository in the U.S. (5) is designed for siting in bedded evaporite deposits. The research results presented here were carried out to address certain intrinsic materials performance and property questions, dictated by the requirements of potentially important applications of cementitious materials where they will perform in the presence of halide phases:

(1) Effects upon the chemical and physical properties of such composites prepared with superplasticizing admixture and salt combinations: Are there potential interactions that would affect the integrity of the composites?

(2) Effects of alkali halides and superplasticizers upon the rheological properties of "pumpable" cementitious slurries: Because cement formulations for down-hole emplacement are usually designed to be pumped under turbulent flow conditions (large Reynold's numbers) it is necessary to determine any such effects.

(3) Interactions of halides with the superplasticizers, that will affect the "pumpability" of the slurries: Do any significant changes occur with time? The purpose of the following experiments was therefore to address the questions of potential interaction between halide phases present in the expected geological environment and the cementitious mixes that might be emplaced in the geomeidia.

## EXPERIMENTAL

Ordinary commercial cements were used in these studies as much as possible, modified by chemical additions to achieve additional desirable chemical and physical properties. The cements used included an ASTM Type I and an API Class C. Table 1 gives chemical analyses of these cements. Three commercial superplasticizers, one sulfonated melamine formaldehyde condensate (A14), and one sulfonated naphthalene formaldehyde condensate (A3), were employed in the studies.

All cement preparations were conducted following ASTM C305 procedures; samples were mixed with either deionized water, or sodium chloride saturated solutions and maintained at a w/c of 0.3, compacted into polyethylene molds on a vibrating table for 60 seconds, and cured for 24 hours at greater than 90% relative humidity (1). The molds were then removed, and the samples were cured under selected fluids, saturated  $\text{Ca}(\text{OH})_2$  or saturated NaCl, contained in sealed glass jars at room temperature and 60°C. The nomenclature used is as follows:

cement type	w/c	mixing water	% admixture	curing water	curing temp.
I	0.3	NaCl*	1	$\text{Ca}(\text{OH})_2$	RT
I	0.3	DW	0	NaCl	60.

\*The concentration of NaCl in the solution is not included as solid in the designated water:solid ratio; DW = deionized water.

	Type I (I-5)	Class C (C-2)
SiO <sub>2</sub>	20.82	21.2
Al <sub>2</sub> O <sub>3</sub>	5.24	2.7
Fe <sub>2</sub> O <sub>3</sub>	2.90	5.1
CaO	62.88	65.4
MgO	3.20	0.8
SO <sub>3</sub>	2.88	2.5
CO <sub>2</sub>	ND	ND

Table 1.

Chemical composition (major constituents) of Type I and Class C cements

Compressive strength (ASTM C109), microhardness (Vicker's), density (1), water and gas permeability (6), bond strength, phase composition by x-ray diffraction, setting times (ASTM C191), and rheological properties (7) were determined to aid in the interpretation of potential interactions.

## RESULTS AND DISCUSSION

These experiments were designed to investigate various properties of cementitious materials relevant to their placement and performance in contact with salt-saturated environments. Initial experiments were designed to compare the effects of deionized mixing water vs. saturated NaCl mixing water; the effect of the interaction of a fixed concentration (1%) of a superplasticizer with NaCl added to the mixing water; the effect of the type of curing water [sat.  $\text{Ca}(\text{OH})_2$  or sat. NaCl], and in a limited number of tests the effects of curing at elevated temperatures.

X-ray Diffraction Phase Determination. Very little difference in the resultant hydration products could be recognized between the samples that were prepared with and without an admixture. The phases identified by x-ray diffraction (XRD) were portlandite [ $\text{Ca}(\text{OH})_2$ ],  $\beta\text{-C}_2\text{S}$ ,  $\text{C}_3\text{S}^*$  and the calcium chloroaluminate hydrate "Friedel's salt" (8). Also recognized in these diffractograms were a large amorphous contribution to the x-ray diffraction pattern centered at  $\sim 25^\circ 2\theta$ , and the contribution of a phase that possesses intense low angle diffuse background which usually left the chart, beginning in the range of from 6 to  $10^\circ$ . Table 2 is a summary of the recognized phases. The present XRD data were obtained for qualitative phase identification; the references therefore necessarily reflect only trends of the reactions.

Phases identified by x-ray diffraction	curing time (days)	admixture (A3)*								
		0%				1%				
0.3 NaCl-Ca(OH) <sub>2</sub> -RT	7	P	B	C	1b	P	B	C	1b	
	28	P	B	C		P	B	C		
	56	P	B	C		k	P	B	C	k
0.3 NaCl-NaCl-RT	7	P	B	C	1b	P	B	C	1b	
	28	P	B	C		P	B	C		
	56	P	B	C		k a	P	B	C	ka
0.3 DW-Ca(OH) <sub>2</sub> -RT	7	P	B	C	1b	P	B	C		
	28	P	B	C		a	P	B	C	a
	56	P	B	C		P	B	C		
0.3 DW-NaCl-RT	7	P	B	C	1b	P	B	C	1b	
	28	P	B	C		k	P	B	C	k
	56	P	B	C		P	B	C		

\*Nomenclature in order: w/c ratio, mixing water, curing water, curing temperature.

P = portlandite, B =  $\beta\text{-C}_2\text{S}$ , C =  $\text{C}_3\text{S}$ ; 1b = low angle background; k = Friedel's salt ( $3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{CaCl}_2\cdot 10\text{H}_2\text{O}$ ); a = 60°C cured sample; a = amorphous background; A3\* = sulfonated naphthalene formaldehyde condensate.

\*Cement abbreviations: C = CaO, S =  $\text{SiO}_2$ , A =  $\text{Al}_2\text{O}_3$ , F =  $\text{Fe}_2\text{O}_3$ .

## VI-172

**Bulk Density.** Bulk density is a routinely measured control property which, when all other factors are equal, confirms reproducibility of sample preparation (i.e., replicates should agree within a high degree of precision). It is also a useful measurement for indicating trends. Therefore, such densities were measured for each sample in this series of experiments, as mass per unit volume, after removal from the curing solution, saturated, surface dry (see Table 3).

The relative comparisons are as follows: (1) With the exception of two compositions which showed significant departures, a specimen of a particular composition was found to maintain its density (within  $\pm 1.5$  percent), not changing with curing time (8 of 12 were within  $\pm 1$  percent). (2) The admixture-containing samples as a group showed the lowest overall variation as a function of time. (3) The de-ionized water preparations showed less variation than the salt-saturated mixing water specimens. (4) Within the deionized mixing water group, samples prepared with admixtures at a single age were essentially identical.

It should be noted that the specimens made with saturated NaCl mixing water actually contain considerably higher total solids content. Thus it had been expected that this group of samples would possess considerably higher densities. In fact, only a single sample set (that with both NaCl mixing and curing water, no admixture, and cured at room temperature) showed considerably higher densities than other comparable samples. Thus, the salt-saturated samples departed significantly from expected ideal behavior.

**Microhardness.** Following earlier work (9) which showed that a relationship exists between microhardness and porosity for certain families of cement-like materials, a relation between microhardness and bulk density was also established (10,11), which is indirectly

related to strength. The microhardness of every sample was determined using the Vicker's technique (10); data are given in Table 4. The general trend found for all samples was an increase in hardness with increasing curing time. The hardness increase ranged from 10% to 70% in some instances with the average increase approximating 30%. The reference neat paste (without admixture or NaCl) cured at 60°C showed the most rapid increase in hardness and also possessed the greatest hardness. Generally the samples prepared with deionized water and Type I cement were harder than those prepared with salt. All of the samples using deionized mixing water produced harder products than those samples prepared with salt mixing water. The former samples generally maintained their strength with respect to the latter samples as a function of time. A very distinct difference in hardness exists between the admixture-containing samples prepared with deionized water and those prepared with saturated sodium chloride. The samples prepared with deionized water are fully 40% stronger than the salt-admixture prepared samples.

**Permeability.** One of the most effective predictive measures of the potential for mass transport through a particular medium is its permeability (6). If a cementitious material is going to be an effective barrier to the transfer of fluids and their contents, it must possess a permeability less than or equal to that of the surrounding rock. The permeability measurements recorded in this experimental sequence were all made with nitrogen, using the Klinkenberg extrapolation to infinite pressure, giving an approximate liquid permeability. The values obtained for the entire specimen set, range from about 0.1 microdarcy to tens of microdarcies. No obvious trend is apparent in these data as a function of the admixture's presence. All those samples prepared with deionized water appear to have permeabilities that are less than or equal to the values obtained for the saturated sodium chloride mixing water samples. The data are reported elsewhere (12).

## OVERVIEW OF PHYSICAL PROPERTIES

Microhardness measurements showed a general increase with curing time, which would be indicative of generally increased strength. The samples prepared with deionized water were generally superior to those prepared with saturated salt solutions. This gives rise to a suggestion that optimized cementing compositions possibly may not involve preparation with saturated salt solutions. On the other hand, samples prepared with deionized water and cured in a 20% salt solution were generally equal to or superior to any of the former samples.

Further, it is realized also that microhardness of the present samples cannot be expected to follow a strictly linear relationship with bulk density for two reasons: (1) The salt-saturated samples cannot be compared strictly with ordinary silicate cement samples because of major compositional difference. NaCl intrinsically has a lower hardness than silicates; although its addition serves to increase density of the composites, it does little to increase the hardness. (2) The density of samples containing only bound (non-evaporable) water would be the more appropriate property for correlation with microhardness, rather than the saturated bulk density, which includes a component of non-structural water. The latter adds little to the strength. Therefore, the above correlation, while showing trends, does not

See Table 3, page VI-175

Table 4. Microhardness vs. curing time for sodium chloride treated Type I pastes

			time (weeks)		
			1	4	8
I-0.3 NaCl-O-Ca(OH) <sub>2</sub> -RT	3A		41.5	53.0	63.8
I-0.3 NaCl-I-Ca(OH) <sub>2</sub> -RT	1A		36.3*	50.7*	50.3*
I-0.3 NaCl-O-Ca(OH) <sub>2</sub> -60	3A'		49.0	63.0	55.2
I-0.3 NaCl-O-NaCl-RT	3B		44.8	50.5	77.0
I-0.3 NaCl-I-NaCl-RT	1B		36.8*	45.3*	52.9*
I-0.3 NaCl-O-NaCl-60	3B'		53.0	60.0	51.4
I-0.3 DW-O-Ca(OH) <sub>2</sub> -RT	4A		59.8	59.1	66.7
I-0.3 DW-I-Ca(OH) <sub>2</sub> -RT	2A		57.1*	69.4*	63.9*
I-0.3 DW-O-Ca(OH) <sub>2</sub> -60	4A'		59.1	81.0	81.8
I-0.3 DW-O-NaCl-RT	4B		55.5	62.6	66.7
I-0.3 DW-I-NaCl-RT	2B		54.1*	63.9*	67.3*
I-0.3 DW-O-NaCl-60	4B'		52.4	66.4	58.7

\*with admixture

constitute a quantitative relationship. It will be important to find whether there are correlations with dry (110°C) density, and to further characterize the cement composites' mechanical properties.

Although general trends can be ascertained that suggest that the sodium chloride based formulations do not form as good a composite as similar formulations without the salt, there was no specific evidence of superplasticizer-salt interactions.

**Rheological Properties.** The questions addressing the potential influence upon the setting times and rheological properties, which affect pumpability do indirectly suggest that interactions occur.

Literature references (13,14) state that in the presence of brines no adverse effects occurred between the saline mixing waters and commercial oil well cementing admixtures. Both of the above references, however, failed to identify the general class of admixtures or to provide any specific details of the nature of any experimentation that was conducted.

All viscosity measurements were made on a Brookfield Model 5X HBT. Other measurements in the laboratory have been carried out with a Haake Rotovisco instrument (7), and calibrations using standard Newtonian liquids have shown agreements in calculated viscosities from the two instruments to within 3-5% (15). The Brookfield instrument was chosen for the current studies for convenience, as well as for the instrument's ability to permit measurement of mortars as well as pastes. The torques at 8 different shear rates were determined in successive order from the largest to the smallest (measurement duration 1 min.), followed by a 1 minute rest, then determined with successively larger shear rates. The total time required for the measurement after mixing was approximately 17 minutes.

Figures 1 and 2 contain data sets for the instrument reading given in millivolts, which is proportional to rheological shear stress, versus added sodium chloride solids in weight percent. The data are graphically presented for one rotor speed (10 rpm), which is proportional to shear rate; this represents an intermediate value of the 8 rates determined. The data in Figure 1 suggest a nearly monotonically decreasing shear stress with increasing salt content (at 1% concentration: wt admixture solids/weight cements, of both the sulfonated naphthalene and melamine formaldehyde condensate superplasticizers). The Type I cement alone undergoes somewhat more erratic behavior but with roughly the same trend. The Class C cement in Figure 2 behaves in a similar fashion with a monotonically decreasing shear stress with increased salt concentrations. The exception to this is the mixture of Class C cement with the sulfonated naphthalene superplasticizer (A3) which is approximately a factor of 3 more fluid than the remaining 2 samples. In both sets of experimental data, erratic behavior occurs in the rheological properties of the pastes below 15 wt/o NaCl.

Typical of the rheological properties with varying superplasticizer concentrations are the data presented in Figure 3, where the shear stresses with increasing superplasticizer concentration generally appear to decrease monotonically. However, in the presence of 25 wt/o sodium chloride, the pastes typically are a factor of 2 to 3 more fluid at low superplasticizer concentrations (less than or equal to 1%) but rapidly

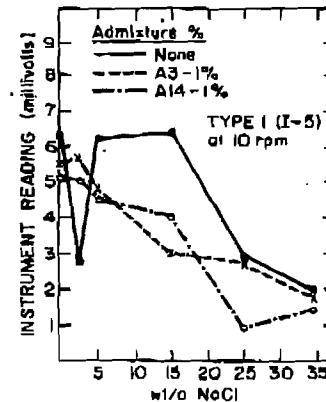


Figure 1. Viscometric response of paste vs. % NaCl in mixing water.

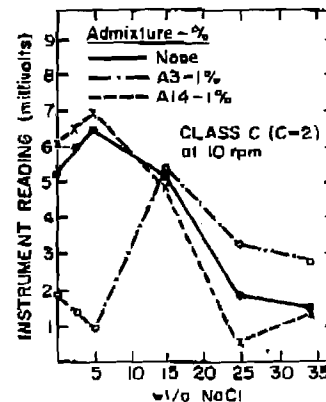


Figure 2. Viscometric response of paste vs. % NaCl in mixing water.

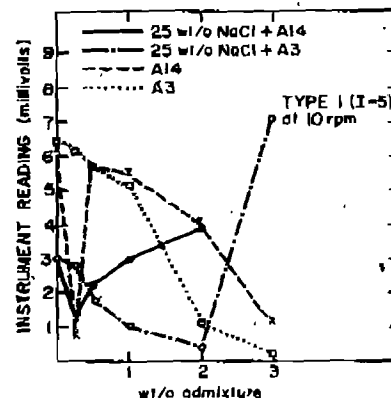


Figure 3. Viscometric response of a Type I (I-5) cement paste to different concentrations of admixtures and NaCl.

change in their fluidity in contact with larger concentrations of either superplasticizer.

**Setting Times.** Extensive measurements were made of setting times of the variety of pastes (1,16). Initial measurements performed on the neat cement Type I (I-5) pastes with increasing admixture concentrations

## VI-174

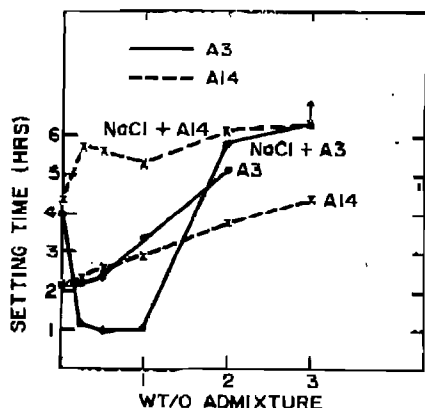


Figure 4. Effects on initial setting time of Type I cement of varying amounts of water-reducing agents in the presence of 25 wt/o NaCl.

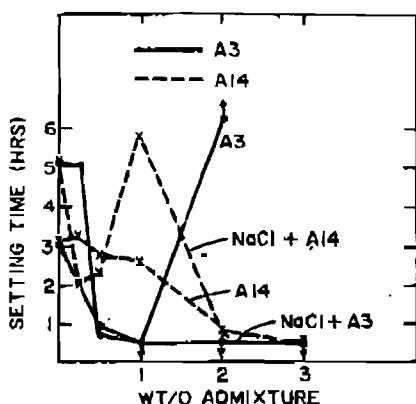


Figure 5. Effects on initial setting time of Class C cement of varying amounts of water-reducing agents in the presence of 25 wt/o NaCl.

indicated a systematic increase in setting times with increasing admixture content. Variations due to the chemical type of admixture were observed especially at the large concentration levels (Figure 4). The Class C (C-2) cement, however, exhibited the opposite trend of setting faster with increased admixture concentrations with very erratic results occurring for the chemically different admixtures at concentrations above 1 wt/o (Figure 5).

The explanation for these observations may lie in a combination of the physical properties of the individual grinds for each cement, and its chemistry. The chemical compositions given in Table 1 show both a lower alumina (and resultant lower  $C_3A$ ) and slightly lower  $SO_3$  content in cement C-2; and C-2 has a slightly higher specific surface area.

The setting times of the Type I (I-5) cement (Figure 4) in the presence of 25 wt/o NaCl generally indicate a trend toward the retardation of setting with increased admixture concentrations. The sulfonated naphthalene formaldehyde condensate A3 behaves erratically, acting as an accelerator at low concentration and as a retarder at higher concentrations. The Class C (C-2) cement exhibited a trend of acceleration of the setting with increased admixture concentrations.

In the four data sets presented, the sulfonated melamine formaldehyde condensate for the most part behaves in a more nearly predictable manner than the sulfonated naphthalene but the Class C (C-2) cement responds in a more predictable manner than the Type I.

Additional experiments with 1% admixture and varying salt concentrations up to saturation further substantiate these observations (16).

#### CONCLUSIONS

Phase characterization of cement pastes prepared with various combinations of ingredients (including presence or absence): mixing water, admixture, curing water; and curing temperature was made. The calcium chloroaluminate hydrate, Friedel's salt, was characteristic of many specimens which either included NaCl in their mix or curing solution. Physical properties studies of the products of cement pastes prepared with superplasticizers in the presence of sodium chloride suggest that the cured products typically are less predictable and often inferior to those of the analogue formulations prepared without the presence of sodium chloride. E.g., these are typified by similar permeability values but with a larger, more erratic scatter in the measured values for the salt-containing samples.

Investigations of the rheological properties of these pastes in the presence of sodium chloride are more revealing experiments. The data presented herein suggest that potential interactions do exist between the superplasticizers (irrespective of the formulation or type of admixture) and salt to produce erratic behaviors. Fortunately, the erratic behavior appears to be limited to low salt concentrations (at low superplasticizer levels) or at relatively high superplasticizer concentrations at more nearly typical working salt-concentrations. This trend, fortunately, will minimize their erratic behavior for certain applications: during emplacement of a slurry the mix most likely will become enriched in salt as it is pumped into an evaporite formation. Data in Figures 2 and 3 would suggest that this circumstance will have little effect upon the rheological properties as compared to the impossible circumstance in which the concentration of admixture increased.

When designing cementitious mixes for geothermal cementing, there would appear to be little advantage, and possible disadvantage, to including salts in the initial mixes (2,3). For plugging applications in evaporites, factors involved with the individual situation would have to be considered. From the present data, either salt-containing mixes or non-salt-containing ones apparently could be employed; but other factors relating to longevity would also have to be considered for their final evaluation (4).

#### ACKNOWLEDGEMENT

The research reported herein was sponsored through subcontracts from the U.S. Department of Energy and administered by the Office of Nuclear Waste Isolation, Battelle Memorial Institute, Columbus, OH.

#### REFERENCES

1. D.M. ROY, B.E. SCHEETZ, E.L. WHITE; and M. DAIMON, "Borehole Cement and Rock Properties Study: Part A, Borehole Plugging Cement Studies," ONWI-5 Annual Progress Report, ONWI/SUB/78/E512-00500, Oct. 30 (1978), 130 pp.

2. D.M. ROY, E.L. WHITE, C.A. LANGTON, and M.W. GRUTZECK, "Potential New High Temperature Cements for Geothermal Wells," Proc. 1979 SPE Intl. Symp. Oilfield & Geothermal Chemistry, Jan. 22-24, 1979, Houston; pp. 153-161.
3. D.M. ROY, E.L. WHITE, C.A. LANGTON, and M.W. GRUTZECK, "New High Temperature Cementing Materials for Geothermal Wells: Stability and Properties," Final Report to DOE, Contract EY-76-C-02-1016, Nov. 30 (1979).
4. D.M. ROY, M.W. GRUTZECK, and P.H. LICASTRO, "Evaluation of Cement Borehole Plug Longevity," Report No. ONWI-30, Topical Report for ONWI (Feb. 1979), 35 pp.
5. U.S. Dept. of Energy, Proc. Natl. Waste Terminal Storage Program Information Meeting, ONWI-62 (Oct. 30, 1979), 232 pp.
6. E.L. WHITE, B.E. SCHEETZ, D.M. ROY, K.G. ZIMMERMAN, and M.W. GRUTZECK, "Permeability Measurements on Cementitious Materials for Nuclear Waste Isolation," pp. 471-478 in Scientific Basis for Nuclear Waste Management, Vol. 1; Proc. Symp. on 'Science Underlying Radioactive Waste Management,' Materials Research Society Annual Meeting, Boston, MA, Nov. 28-Dec. 1, 1978; Ed. G.J. McCarthy.
7. D.M. ROY and K. ASAGA, "Rheological Properties of Cement Mixes. III. The Effects of Mixing Procedures on Viscometric Properties of Mixes Containing Superplasticizers," Cem. Concr. Res. 9 (6), 731-740 (1979).
8. H.G. SMOLCZYK, "Chemical Reactions of Strong Chloride-Solutions with Concrete," Proc. Fifth Intl. Symp. Chem. Cement, Tokyo, Vol. III, pp. 274-280 (1968).
9. I. SOROKA and P.J. SEREDA, "The Structure of Cement-Stone and the Use of Compacts as Structural Models," Proc. Fifth Intl. Symp. Chem. Cement, Tokyo, Vol. III, pp. 67-73 (1968).
10. G.R. GOUDA, "Preparation and Characterization of Very High Strength Hot Pressed Cement Pastes," PhD Thesis, The Pennsylvania State University (August 1975); also G.R. GOUDA and D.M. ROY, Cem. Concr. Res. 5, 551-564 (1975).
11. S.O. OYEFESEBI, "Novel Processing of Cement Phase: A Comparison," PhD Thesis, The Pennsylvania State University (1976); also S.O. OYEFESEBI and D.M. ROY, Cem. Concr. Res. 2, 165-172 (1977).
12. B.E. SCHEETZ, D.M. ROY, E.L. WHITE, and D. WOLFE-CONFER, "Comparison of Tailored Cement Formulations for Borehole Plugging in Crystalline Silicate Rocks and Evaporite Mineral Sequences," Proc. Symposium on 'Science Underlying Radioactive Waste Management,' Materials Research Society Annual Meeting, Boston, MA, Nov. 1979 (in press).
13. K.A. SLAGLE and D.K. SMITH, "Salt Cement for Shale and Bentonitic Sands," J. Petrol. Tech. 187-194 (1963).
14. W.C. CUNNINGHAM and D.K. SMITH, "Effect of Salt Cement Filtrate on Subsurface Formation," J. Petrol. Tech. 239-263 (1968).
15. K. ASAGA, unpublished work.
16. D.M. ROY, P.H. LICASTRO, B.E. SCHEETZ, E.L. WHITE, M.W. GRUTZECK, and K. ASAGA, "Borehole Cement and Rock Properties Study, Task I: Borehole Plugging Cement Studies," ONWI/SUB/78/E512-00500-2 (Task I), Quarterly Progress Report (March 30, 1979).

Table 3. Bulk density of sodium chloride treated samples versus curing time

			admixture					
			1 week		4 weeks		8 weeks	
			without	with	without	with	without	with
I-0.3NaCl-0-Ca(OH) <sub>2</sub> -RT	3A	1.94			1.91		1.94	
I-0.3NaCl-1-Ca(OH) <sub>2</sub> -RT	1A			1.88		1.85		1.88
I-0.3NaCl-0-Ca(OH) <sub>2</sub> -60	3A'	1.87			1.88		1.87	
I-0.3NaCl-0-NaCl-RT	3B	2.07			1.95		2.02	
I-0.3NaCl-1-NaCl-RT	1B			1.93		1.87		1.88
I-0.3NaCl-0-NaCl-60	3B'	1.94			1.96		1.90	
I-0.3DW-0-Ca(OH) <sub>2</sub> -RT	4A	1.89			1.88		1.90	
I-0.3DW-1-Ca(OH) <sub>2</sub> -RT	2A			1.90		1.90		1.91
I-0.3DW-0-Ca(OH) <sub>2</sub> -60	4A'	1.86			1.90		1.89	
I-0.3DW-0-NaCl-RT	4B	1.94			1.91		1.91	
I-0.3DW-1-NaCl-RT	2B			1.91		1.90		1.91
I-0.3DW-0-NaCl-60	4B'	1.92			2.00		1.93	
Average (kg/dm <sup>3</sup> )			1.93±0.07		1.91±0.04		1.92±0.05	
			1.91±0.02		1.88±0.02		1.90±0.02	